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Irreversible Volume Expansion of a TATB-Based Composite and Compressive Strength

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Abstract. It has long been known that compacted composites containing TATB (triaminotrinitrobenzene) crystals undergo "ratchet growth," an irreversible volume expansion upon thermal cycling. A clear mechanism has not been established for this phenomenon, but is believed to arise from the highly-anisotropic CTE of TATB crystals and interactions caused by compaction. Explosive performance depends fundamentally on bulk density, so the effect is important. PBX 9502 is a plastic bonded explosive containing 95 wt% TATB crystals. We have monitored uniaxial length changes of PBX 9502 specimens for various thermal cycles providing mechanistic insight. Post-cycled specimens were compression tested to determine if mechanical properties correlated with the detailed thermal history.

INTRODUCTION

When compared with other explosives, significantly reduced sensitivity to non-shock initiation makes TATB and its plastic-bonded composites ideal for many applications [1]. A potential concern with compactions of TATB-based explosives is their propensity for irreversible volume expansion (ratchet growth) upon thermal cycling. Recent work [2] has demonstrated that the plate-like structure of TATB crystals causes them to align according to consolidation forces, resulting in TATB texture in the solid PBX and anisotropy in the coefficient of thermal expansion (CTE) and in the ratchet growth response.

By controlling compaction/orientation, we have controlled the TATB texture in PBX 9502 specimens and initiated a careful ratchet growth characterization study. We have explored the effects of texture, thermal range, and number of cycles, and have demonstrated that irreversible volume expansion occurs during extended isothermal conditions following a temperature excursion [3]. We here report recent results in our characterization study of the irreversible volume expansion of PBX 9502. In addition to mapping out expansion caused by thermal cycling, we have performed post-cycle compression tests to learn if strength depends on density alone or varies according to thermal profile details.

EXPERIMENTAL

PBX 9502 is 95 wt% TATB crystals with 5 wt% Kel-F binder. The PBX 9502 specimens in this study were 5 mm diameter x 5 mm length cylinders machined from a large isostatic-pressed hemispherical charge. Specimens are from radially-symmetric locations and orientations, shown previously to be equivalent in texture [2] and density as well [4]. Immersion methods for density measurement are not reliable for such small specimens; using measured dimensions and mass, the densities were determined to be 1.90 g/cm^3.

Ratchet grown tests were performed on a thermal mechanical analyzer (TMA), model Q400 by TA Instruments. Thermal profiles all started and ended at 23°C and ramped to X°C with a rate of 1°C/min. Once at X°C, the temperature was held for 10 hrs in one series of tests, and 10 min in another, before returning back to 23°C where a 10-min soak was held, followed by another thermal cycle. In some cases, thermal cycles were repeated, in other cases, thermal cycles were mixed (different X°C values). The TMA varies the temperature and measures the specimen length as a function of time under a constant force of 0.3N. Thermal cycling of dry-pressed TATB confirms that TATB is the PBX 9502 component responsible for ratchet growth (data not shown); thermal cycling of mock PBX (no TATB but the same Kel-F binder), shows compressive creep under the 0.3N of load (i.e. linear contraction instead of expansion). We have also thermal cycled PBX 9502 specimens cut perpendicular to the specimens from this study, confirming texture anisotropy [2]. Due to manuscript length restrictions, we do not include these data here.

Quasi-static uniaxial compression tests were performed on a subset of post-thermal cycled PBX 9502 specimens. These were tested on an Instron 5567 with an attached Bemco environmental chamber. All tests were run at 23°C and 0.05in/min crosshead speed. Engineering stress was measured as a function of engineering strain, where the latter was deduced using crosshead displacement.

Post thermal-cycling specimen densities were estimated using the final ambient axial strain measurements from the TMA. Using the ratchet growth anisotropy as measured by us for similarly prepared and oriented specimens [5], we calculated a final ratchet grown volume and density.

RESULTS AND DISCUSSION

Figure 1 is an example of TMA data. The test shown alternates thermal cycles between X = -43°C and 113°C with 0.17-hr holds at X and also whenever 23°C is encountered. The 0.17-hr holds at 23°C allow us to measure the equilibrated strain at this temperature before and after each cycle to X. Using the strain values measured at 23°C, we calculate the Δε growth for hot cycles and for cold cycles (see inset).

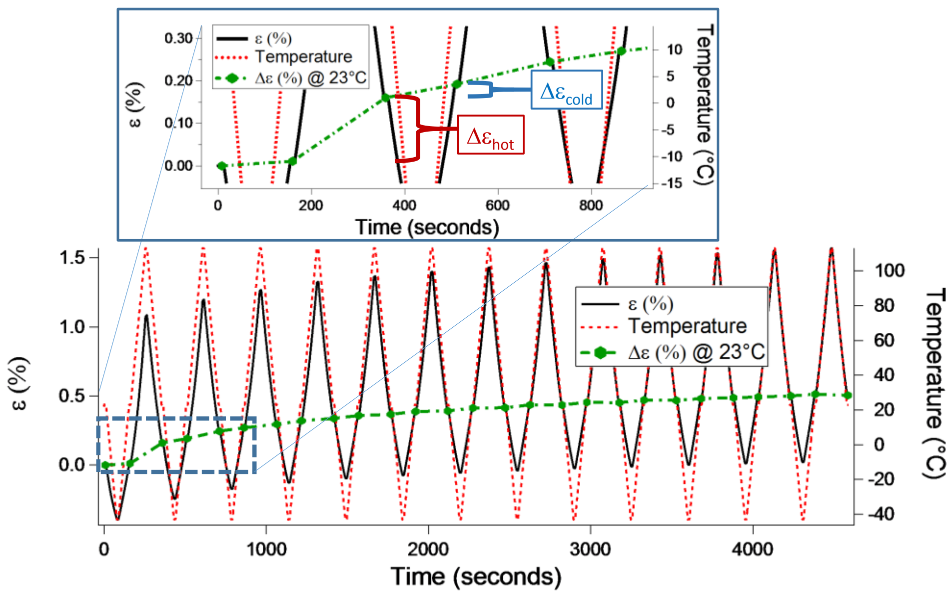

FIGURE 1: TMA data, temperature and strain versus time, where X alternates between -43°C and 113°C; green circles (dotted line) show ε values at 23°C. The inset zooms in on the ε values at 23°C for the first six cycles, cold alternating with hot.

The $\Delta\varepsilon_{hot}$ and $\Delta\varepsilon_{cold}$ values obtained from Fig 1 are plotted versus cycle number in Fig 2a, and versus the respective accumulated strains, $\Sigma\Delta\varepsilon_{hot}$ and $\Sigma\Delta\varepsilon_{cold}$ in Fig 2b. Data from the alternating -43 and 113°C cycles are plotted with open symbols. Additionally, on each of these graphs we plot Δε values from specimens that were cycled repeatedly to a single X temperature (solid symbols). Specifically, we show data for tests with X = -43°C and for X = 113°C.

In Fig 2a, for the test with X = 113°C (red, solid), the first cycle $\Delta\varepsilon_{hot}$ is large (0.20%) and drops significantly by the second cycle. Subsequent cycles show diminishing $\Delta\varepsilon_{hot}$. For the test with X = -43°C, the first cycle $\Delta\varepsilon_{cold}$ is very small (0.02%) and diminishes further with subsequent cycles. However, for the tests where cycles alternate between X = -43 and 113°C (open symbols), note that there is an increase in the value of $\Delta\varepsilon_{cold}$ for the second cycle, and that the values are relatively constant for the next 10+ cycles. There is a definite "boost" to the cold cycle growth for having cycled to warm in between. Likewise, there is a slight increase in the $\Delta\varepsilon_{hot}$ values for the second, third and fourth cycles (the first three hot cycles that follow a cold cycle in the test series); the “boost” is smaller here, but the first few hot cycles grow more when they are cold cycled in between.

In Fig 2b, the exact same $\Delta\varepsilon_{cold}$ and $\Delta\varepsilon_{hot}$ data are plotted versus their respectively accumulated strains and we see how the per cycle strains contribute to the overall specimen expansion. Here, for the alternating cold-hot cycles (open symbols), the accumulated cold strain ($\Sigma\Delta\varepsilon_{cold}$) after 10+ cycles is 0.23%, nearly as large as the accumulated hot strain ($\Sigma\Delta\varepsilon_{hot}$) after 10+ cycles (0.32%); cold cycles, when alternated with warm cycles, contribute a nearly constant value of $\Delta\varepsilon_{cold}$ for all 10 cycles, whereas the hot cycles, alternated with cold, contribute large $\Delta\varepsilon_{hot}$ values initially that drop off very quickly. In contrast, when cycles are not alternated between hot and cold, the accumulated cold strain ($\Sigma\Delta\varepsilon_{cold}$) after 10+ cycles is only 0.05%, while the accumulated hot strain ($\Sigma\Delta\varepsilon_{hot}$) after 10+ cycles is 0.29%. We conclude that there is a significant increase in the irreversible expansion of cold cycles *when they are alternated with warm cycles*.

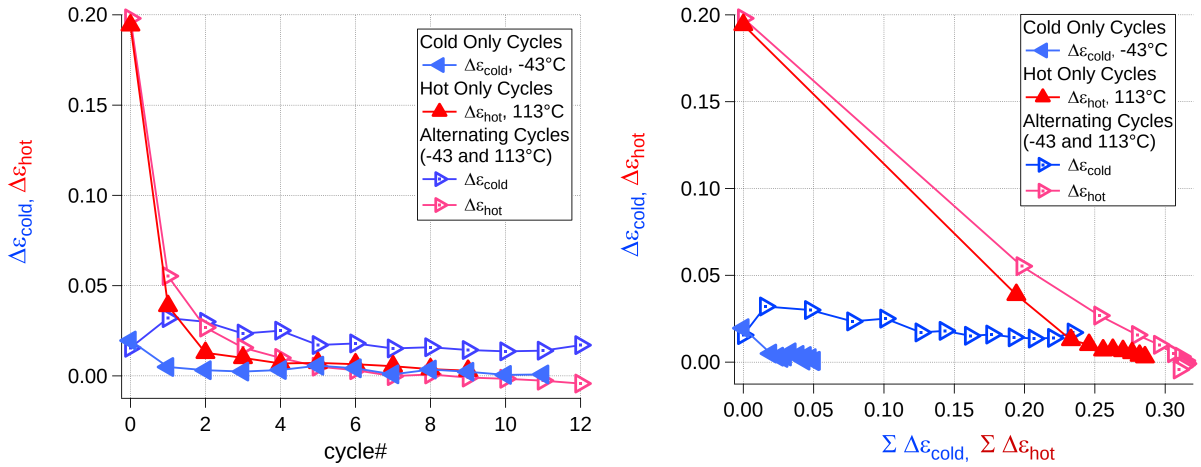

FIGURE 2: $\Delta\varepsilon_{hot}$ and $\Delta\varepsilon_{cold}$ values plotted versus (a) cycle number and (b) accumulated strains ($\Sigma\Delta\varepsilon_{hot}$ and $\Sigma\Delta\varepsilon_{cold}$, respectively); see legends for test descriptions. Hold times at all X°C were 10 min for these tests.

In Fig 3 values of $\Delta\varepsilon_{hot}$ versus $\Sigma\Delta\varepsilon_{hot}$ are plotted for a different set of tests. Here we show the results for three different tests with repeat cycles to X = 73°C, X = 113°C and 153°C, respectively (solid symbols). We also overlay the results of mixed hot cycles (different X temperatures but all above ambient). For mixed cycles, all hot, we see that the $\Delta\varepsilon_{hot}$ magnitude of a given cycle depends on the temperature (i.e. the “trajectory” defined by cycles to that particular X value), and it depends on where you are along the accumulated strain ($\Sigma\Delta\varepsilon_{hot}$) of that trajectory.

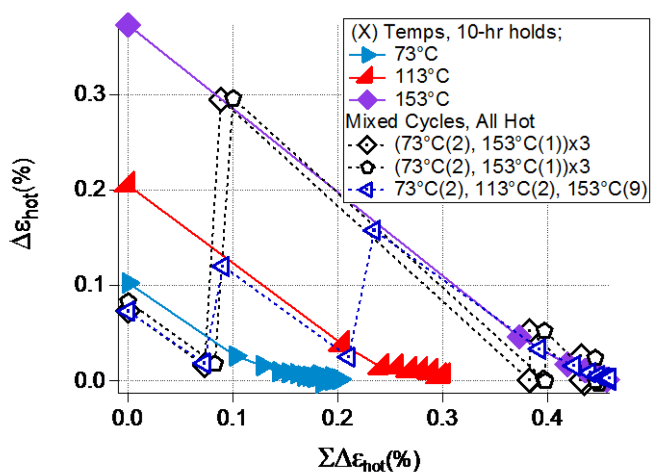

FIGURE 3: $\Delta\varepsilon_{hot}$ values plotted versus accumulated strain $\Sigma\Delta\varepsilon_{hot}$; see legend for test descriptions. Hold times at all X°C were 10 hr for these tests.

It had been hypothesized that the post-thermal-cycled PBX 9502 microstructure might be sensitive to details of the thermal profile: ratchet grown specimens whose porosity increases came from hot cycles only (thermal expansion of compacted crystals) might be expected to differ from those whose expansion came from alternating hot and cold (expansion and contraction). In Fig. 3a below, engineering stress and strain curves are plotted for a subset of the compression tests on post-thermal-cycled specimens from Table 1. The stress maximum and the corresponding strain value are plotted versus estimated ratchet grown density in Fig 3b. Specimens from hot only thermal cycles (solid symbols) and specimens whose cycles alternated cold and hot (open symbols) fall on the same line. If there are microstructural differences for alternating hot-cold cycles, compared to all hot cycles, then they do not play a dominant role in determining compression properties. There is some likelihood that tensile properties would be more sensitive to thermally-induced microstructure differences, although tensile tests on these materials are more difficult to perform.

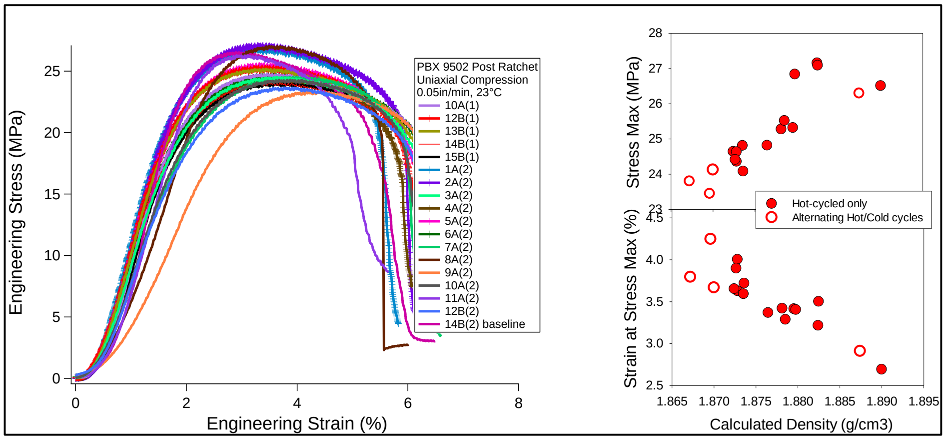

FIGURE 4: (a) Engineering stress and strain curves for select post-thermal-cycled specimens; (b) Compression parameters plotted versus estimated post-thermal-cycled specimen density; specimens thermal cycled hot only have filled symbols, those cycled both hot and cold have open symbols.

CONCLUSIONS

Recent TMA experiments on PBX 9502 specimens with controlled TATB texture have demonstrated that irreversible volume expansion to different X endpoint temperatures is additive for cycles above ambient, however, when cycles alternate between X temperatures above and below ambient, the irreversible growth is not additive. Cold-going cycles show significantly more expansion for repeated thermal cycles if they follow a hot cycle. The details of this observation are being used to inform and calibrate a ratchet growth model. Any microstructural differences that may be caused by differences in thermal profiles do not significantly effect compressive strength or ductility.

ACKNOWLEDGMENTS

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